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(54) **Fluorochemical surfactants and process for preparing same.**

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## Description

This invention relates to fluorochemical surfactants and to a process for preparing such fluorochemical surfactants.

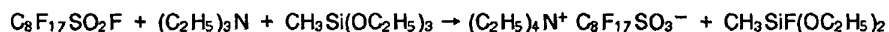
5 Fluorochemical surfactants are well-known for their ability to provide improved wetting, spreading, and leveling in a wide variety of systems. Some typical application areas include floor polishes, alkaline cleaners, leak-detection solutions, corrosion inhibitors, specialty inks, and in processes varying from photographic development and electroplating to the manufacture of clay refractories.

Among the early fluorochemical surfactants are the fluorocarbon dialkylamines and their corresponding  
10 quaternary ammonium derivatives disclosed in U.S. Patent No. 2,759,019 (Brown et al.), the perfluoroalkanesulfonamido alkylenemonocarboxylic acids disclosed in U.S. Patent No. 2,809,990 (Brown), and the N-perfluoroalkylsulfonamidoalkyl halomethylethers and quaternized derivatives thereof disclosed in U.S. Patent No. 3,147,064 (Brown et al.).

Later, U.S. Patent No. 4,006,064 (Niederprum et al.) disclosed, as a fluorine-based surfactant, a  
15 quaternary ammonium perfluoroalkane sulfonate of the formula  $[R_1SO_3]^- [NR^1R^2R^3R^4]^+$  in which  $R^1$ ,  $R^2$ ,  $R^3$ , and  $R^4$  each individually is alkyl, alkenyl, cycloalkyl, or aralkyl of up to 18 carbon atoms, or two or three of  $R^1$ ,  $R^2$ ,  $R^3$ , and  $R^4$  together with the nitrogen atom to which they are attached form a heterocyclic ring, and  $R_1$  is a perfluorinated alkyl radical with 6 to 12 carbon atoms. Niederprum et al. state that this surfactant is useful in electrodeposition of a chrome layer wherein the chrome is deposited from an electrolyte solution  
20 containing a hexavalent chromium compound.

U.S. Patent No. 4,028,257 (Thompson) discloses a perfluorinated compound represented by the formula  $[C_8F_{17}SO_2NHC_3H_5N(CH_3)_3]^+ A^-$  wherein  $A^-$  is  $Cl^-$ ,  $I^-$ ,  $F^-$ , or  $Br^-$ , which is used together with an adduct of trimethyl-1-heptanol plus seven moles of ethylene oxide to lower the surface tension of various aqueous-based liquids, e.g. acids, spent acids, brines, water, etc. and corresponding gelled liquids, when such fluids  
25 are employed in environments where they come in contact with earthen formations.

U.S. Patent No. 4,168,277 (Mitschke et al.) discloses a process for the production of tetraethyl ammonium perfluoroalkyl sulphonates  $(C_2H_5)_4N^+ R_1SO_3^-$ , wherein  $R_1$  represents a perfluorinated alkyl radical with about 4 to 10 carbon atoms, by reacting crude perfluoroalkyl sulphonic acid fluoride which has not been especially purified, with triethylamine and an ethoxy silane in an inert solvent at temperatures of  
30 about 10° to 60°C. Mitschke et al. show, for example, the following reaction equation



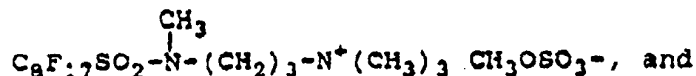
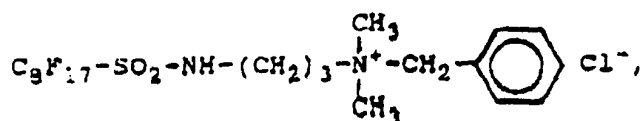
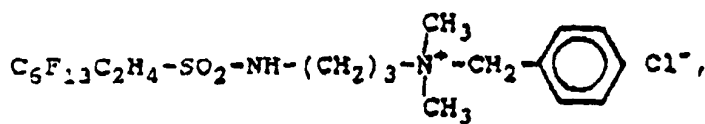
These tetraethyl ammonium perfluoroalkyl sulphonates are described as being useful for suppressing  
35 chromium mist in electro-chromium plating, as an internal mold-release agent in the injection molding of thermoplasts, as wetting agents in the acid polishing of glass and in photographic emulsions.

U.S. Patent No. 4,203,850 (Wirtz et al.) disclose fluorochemical surfactants which have the formula  $R_1CF=CH-CH_2-(^+N-R_4R_5R_6)$  in which  $R_1$  is a perfluoroalkyl group having from 3 to 16 carbon atoms,  $R_4$  is a hydrogen atom, a  $C_{1-4}$  alkyl, cyclohexyl or 2-hydroxyalkyl having from 2 to 6 carbon atoms,  $R_5$  is a  $C_{1-4}$   
40 alkyl radical, a cyclohexyl radical or 2-hydroxyalkyl having from 2 to 6 carbon atoms, and  $R_6$  is a water-solubilizing radical, -Q- $R_7$  with Q being an alkylene group having from 2 to 6 carbon atoms and  $R_7$  being a water-solubilizing polar group such as  $-CO_2^-$ ,  $-SO_2^-$ ,  $-SO_3^-$ ,  $OP(H)O_2^-$ , and especially  $OSO_3^-$ . These surfactants are described as being useful in foam extinguishing agents.

U.S. Patent No. 4,222,828 (Zuurdeeg) discloses a process for electro-codepositing inorganic particles  
45 and a metal on a surface which utilizes fluorochemical surfactants having the structural formulae

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U.S. Patent No. 4,313,978 (Stevens et al.) discloses a method of treating a surface to reduce the static charging thereon by applying an antistatic composition comprising a fluorinated anionic surfactant which is an amino salt of an acid containing a fluorinated organic radical and an antistatic agent which is an ionic salt of an amine. The preferred surfactants are depicted by the formula  $\text{R}_f\text{AX}^-\text{Z}^+$  in which  $\text{R}_f$  represents a fluorinated organic radical, A represents a bond or a divalent linking group,  $\text{X}^-$  represents an acid anion, and  $\text{Z}^+$  represents a quaternary ammonium cation.

U.S. Patent No. 3,723,512 (Niederprum et al.) discloses fluorine-containing quaternary ammonium salts of the general formula

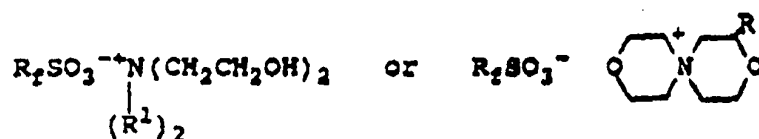


wherein  $\text{R}^1$ ,  $\text{R}^2$ ,  $\text{R}^3$  and  $\text{R}^4$ , which may be the same or different, are alkyl, alkenyl, cycloalkyl or aralkyl radicals or two or three of  $\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  together with the nitrogen may form a heterocyclic ring and  $\text{R}_f$  represents a perfluorinated straight chain or branched aliphatic radical having from one to 12 C atoms. Niederprum et al. also disclose a process for preparing these quaternary ammonium salts characterized in that a perfluorinated sulfonyl fluoride of the formula  $\text{R}_f\text{SO}_2\text{F}$  is reacted in the presence of a tertiary amino with a silicic acid ester of the general formula  $\text{R}_n^5\text{Si}(\text{OR}^4)_{4-n}$  wherein  $\text{R}^5$  represents an aliphatic or an aromatic hydrocarbon radical, and  $\text{R}^4$  represents an alkyl, alkenyl, cycloalkyl or aralkyl radical, and n has a value of from 0 to 3.

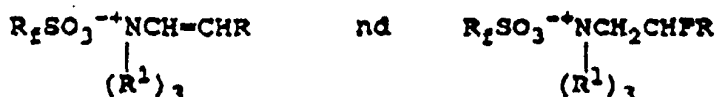
Although the above-discussed fluorochemical surfactants are generally useful in the systems for which they are selected, there has been a need for fluorochemical surfactants which have improved surface tension depressant properties and which can be easily and economically produced.

The present invention provides fluorochemical surfactant compositions which have useful surface tension depressant properties and a method for producing such fluorochemical surfactant compositions easily and economically.

The fluorochemical surfactant compositions of the present invention comprise (a) at least one fluorochemical amine salt which can be represented by the formula:

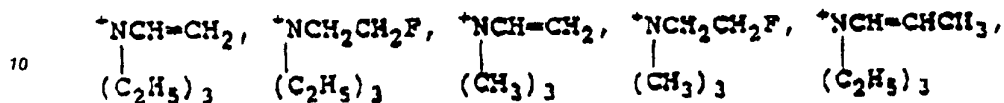


or (b) a mixture of fluorochemical amine salts which can be represented by the formulas

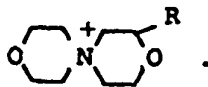


wherein R is H or CH<sub>3</sub>, each R<sup>1</sup> is independently an alkyl radical containing 1 to 4 carbon atoms and may also contain a hydroxyl group, and R<sub>f</sub> is a perfluoroaliphatic radical containing 3 to 20, preferably 6 to 10, carbon atoms and can be straight chain, branched chain, or, if sufficiently large, cyclic, or combinations thereof, such as alkylcycloaliphatic radicals, and which can include catenary oxygen, hexavalent sulfur, and/or trivalent nitrogen hetero atoms bonded only to carbon atoms.

Typical cations of the salts of this invention include, for example,



<sup>+</sup>N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>4</sub>, and

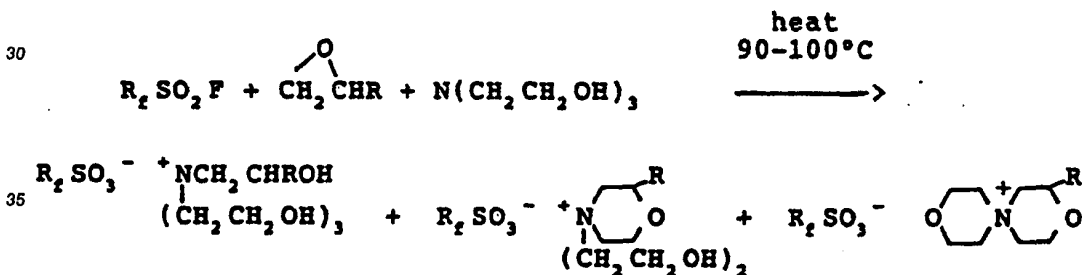
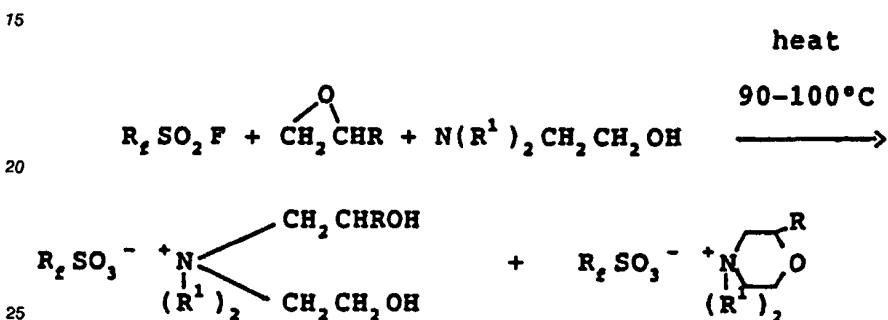
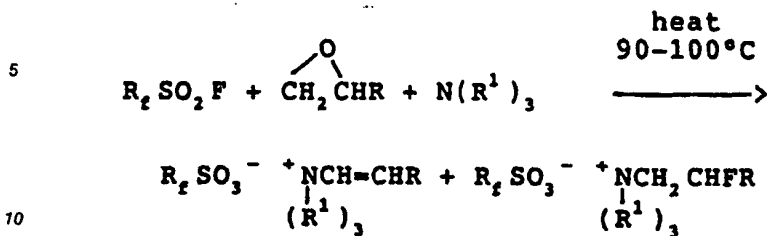


The skeletal chain in the fluoroaliphatic radical can include catenary oxygen, hexavalent sulfur, and/or trivalent nitrogen hetero atoms bonded only to carbon atoms, such hetero atoms providing stable linkages between fluorocarbon portions of R<sub>f</sub> and not interfering with the inert character of the R<sub>f</sub> radical. The fluoroaliphatic radical, R<sub>f</sub>, generally is a fluorinated, stable, inert, non-polar, preferably saturated, monovalent moiety which is both oleophobic and hydrophobic. Preferably R<sub>f</sub> is C<sub>n</sub>F<sub>2n+1</sub>· where n is 6 to 10. The fluorochemical surfactant compositions may also contain small amounts of fluoroaliphatic sulfonate salts having unknown cationic structures.

These fluorochemical surfactant compositions are useful as wetting agents, dispersing agents, foaming and defoaming agents, and antistatic agents, and are particularly useful as surfactants and emulsifiers.

The method of producing fluorochemical surfactant compositions of the present invention comprises reacting at least one perfluoroalkyl sulfonyl fluoride having 3 to 20 alkyl carbon atoms, preferably perfluorooctane or perfluorodecane sulfonyl fluoride, ethylene oxide, or propylene oxide, and at least one tertiary amine, (R<sup>1</sup>)<sub>3</sub>N, wherein R<sup>1</sup> is an alkyl radical having 1 to 4 carbon atoms and which may contain a hydroxyl group. Preferably, the reaction is carried out at a temperature of about 60 to 150°C, more preferably about 90 to 100°C, for a period of about 2 to 16 hours.

This reaction can be represented by the following reaction schemes:



40 where R<sup>1</sup> and R<sub>f</sub> are as previously defined and R is H or CH<sub>3</sub>.

This invention will now be illustrated by the following non-limiting examples.

#### Example 1

45 Into a 350 mL pressure vessel were charged, 48.5 g (0.1 mole) perfluorooctane sulfonyl fluoride, 4.41 g (0.1 mole) ethylene oxide, and 10.12 g (0.1 mole) triethylamine. The vessel was closed and heated to 90 °C with agitation for 12 hours. The vessel was maintained at 90 °C with continuous agitation. The vessel was cooled and the reaction product was dissolved in methylene chloride. The crude yield was 98%. The solution was washed with a small amount of water and dried. The resulting fluorochemical surfactant product was a yellow waxy solid obtained at a yield of 70%. Analysis of the product by H and F nuclear magnetic resonance (NMR) and fast atom bombardment mass spectroscopy confirmed the presence of

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21 mole % of fluoroaliphatic sulfonate amine salts having unknown cationic structure.

#### Example 2

A fluorochemical surfactant was prepared as in Example 1 using as reactants 48.5 g (0.1 mole) perfluorooctane sulfonyl fluoride, 4.41 g (0.1 mole) ethylene oxide, and 5.91 g (0.1 mole) trimethylamine with the reaction temperature being held at 90°C for 7 hours. The reaction product was cooled and dissolved in about 100 mL methanol. The crude yield was 77%. The methanol was evaporated to yield a brown waxy solid. Analysis of the product by H and F NMR confirmed the presence of



10 mole % of fluoroaliphatic sulfonate amine salts having unknown cationic structure.

#### Example 3

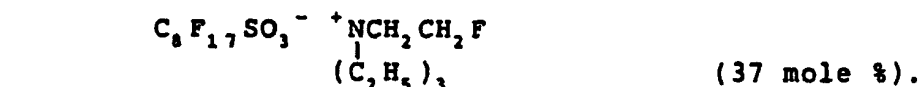
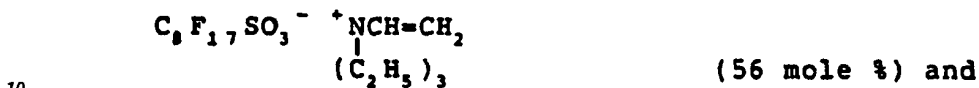
A fluorochemical surfactant was prepared as in Example 1 using as reactants 146.25 g (0.25 mole) perfluorodecane sulfonyl fluoride, 11.01 g (0.25 mole) ethylene oxide, and 25.3 g (0.25 mole) triethylamine with the reaction temperature being 100°C and the reaction continuing for 12 hours. The reaction product was cooled and 178 g (97.5% yield) crude product was collected. Twenty grams of the crude product were dissolved in 30 mL xylene hexafluoride with heating and this solution was washed with water. Phase separation was carried out and the organic phase was dried over magnesium sulfate. The solvent was stripped off and 16 g (78% yield) of a waxy tan solid was obtained. Analysis of the product by H and F NMR confirmed the presence of



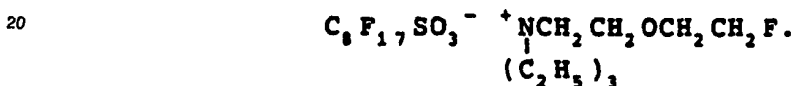
#### Example 4

A fluorochemical surfactant was prepared as in Example 1 using as reactants 121.25 g (0.25 mole) perfluorooctane sulfonyl fluoride, 44.04 g (1.0 mole) ethylene oxide, and 25.33 g (0.25 mole) triethylamine

with the reaction temperature raised to 100°C and held at 100°C for 16 hours. The reaction product was cooled and 187 g (98% yield) of a thick brown product was recovered. Twenty-five grams of the product was dissolved in 40 mL methylene chloride and the solution was washed with water. The product was dried and the solvent was stripped off to yield a waxy brown product (18.2 g, 71% yield). Analysis of the major product by H and F NMR and liquid chromatography confirmed the presence of

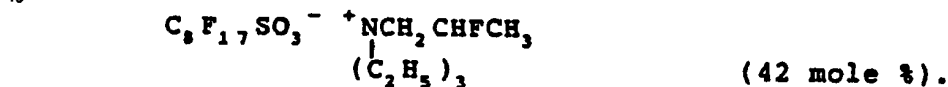
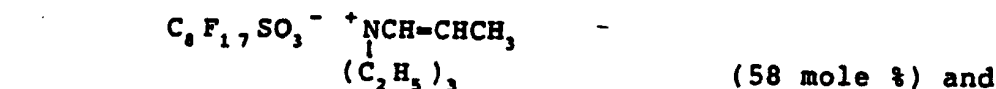


Analysis also showed about 7 mole % of minor products including



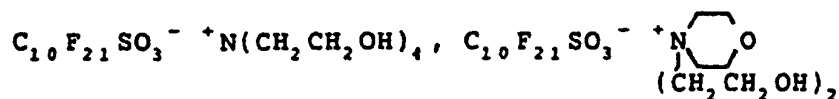
#### Example 5

A fluorochemical surfactant was prepared as in Example 1 using as reactants 121.5 g (0.25 mole) perfluorooctane sulfonyl fluoride, 14.5 g (0.25 mole) propylene oxide, and 25.33 g (0.25 mole) triethylamine with the reaction temperature being raised to 100°C and then held at 100°C for 30 hours. The reaction mixture was cooled and 159 g (99% yield) of a dark viscous product was recovered. Twenty grams of the product was dissolved in 40 mL methylene chloride, washed with water, and dried. After solvent stripping, 14.7 g (73% yield) of a waxy brown product was recovered. Analysis of the product by H and F NMR confirmed the presence of

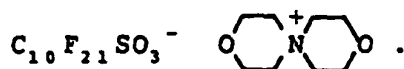


#### Example 6

A fluorochemical surfactant was prepared as in Example 1 using as reactants 146.25 g (0.25 mole) perfluorodecane sulfonyl fluoride, 11.01 g (0.25 mole) ethylene oxide, and 37.3 g (0.25 mole) triethanolamine with the reaction temperature being 95°C and the reaction continuing for 8 hours. The reaction product was cooled and 193 g (99% yield) crude product was collected. 10 g of the crude product was dissolved in methanol with heating and a small amount of product formed a white precipitate. The precipitate was filtered off yielding 0.6 g (6%) of a white, hard, waxy material. The methanol was stripped off the solvent-soluble portion and 7.9 g (78% yield) of a yellow, sticky wax was obtained. Analysis of the yellow wax product by H and F NMR and fast atom bombardment mass spectroscopy indicated the presence of



and a structure containing spiromorpholine cations,



#### Surface Tension Evaluation

Aqueous solutions containing 50 ppm, 100 ppm, 200 ppm, and 400 ppm of each of the above-prepared fluorochemical surfactant compositions were prepared and tested for static surface tension at equilibrium at 22 °C using a Surface Tensiometer™, Model 21 available from Fisher Scientific Co. The results are set forth in Table I. For Comparative Examples C1-C3, fluorochemical surfactants  $\text{C}_8\text{F}_{17}\text{SO}_3^- \text{NH}_4^+$ , and  $\text{C}_8\text{F}_{17}\text{SO}_3^- \text{H}_2\text{N}(\text{C}_2\text{H}_4\text{OH})_2^+$  and  $\text{C}_8\text{F}_{17}\text{SO}_3^- \text{N}(\text{C}_2\text{H}_5)_4^+$  (Example 1 of U.S. Patent No. 4,006,064), respectively, were tested for static surface tension at equilibrium in the same manner as the fluorochemical surfactant of Example 1. For Comparative Example C4, a stock solution was prepared using 4 parts of a 25 weight percent solution of  $\text{C}_{10}\text{F}_{21}\text{SO}_3^- + \text{NH}_4$  in 50:50 by weight of ethyleneglycol monobutyl ether and water, 4 parts dipropylene glycol monomethyl ether, and 92 parts deionized water and aqueous solutions containing 50 ppm, 100 ppm, 200 ppm, and 400 ppm  $\text{C}_{10}\text{F}_{21}\text{SO}_3^- + \text{NH}_4$  were prepared using the stock solution and tested for static surface tension at equilibrium. The results are set forth in Table 1.

Table I

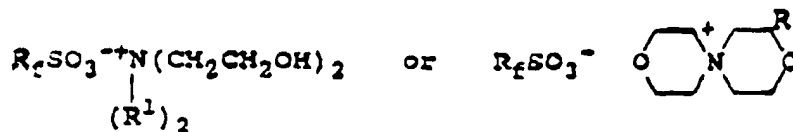
Surfactant of Example Number	Static Surface Tension (dynes/cm @ conc.)			
	50 ppm	100 ppm	200 ppm	400 ppm
1	31.0	23.5	20.5	20.5
2	32.0	24.5	22.5	19.5
3	24.6	20.9	19.3	18.8
4	34.6	31.8	29.3	22.2
5	40.1	34.2	26.8	20.9
6	25.0	21.3	19.5	18.7
C1	47.0	37.5	32.0	26.5
C2	44.0	36.5	27.5	24.0
C3	46.5	39.5	30.0	27.5
C4	32.0	24.0	22.6	21.3

As can be seen from the data in Table I, the fluorochemical surfactant compositions of the invention provide a greater reduction in surface tension than do the comparative fluorochemical ammonium or amine perfluoroalkane sulfonate salt surfactants having the same number of carbon atoms in the sulfonate anion at comparable concentration levels.

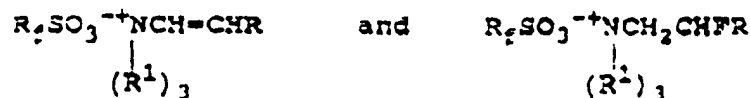
#### Claims

1. Fluorochemical surfactant compositions comprising (a) at least one fluorochemical amine salt which can be represented by the formula:



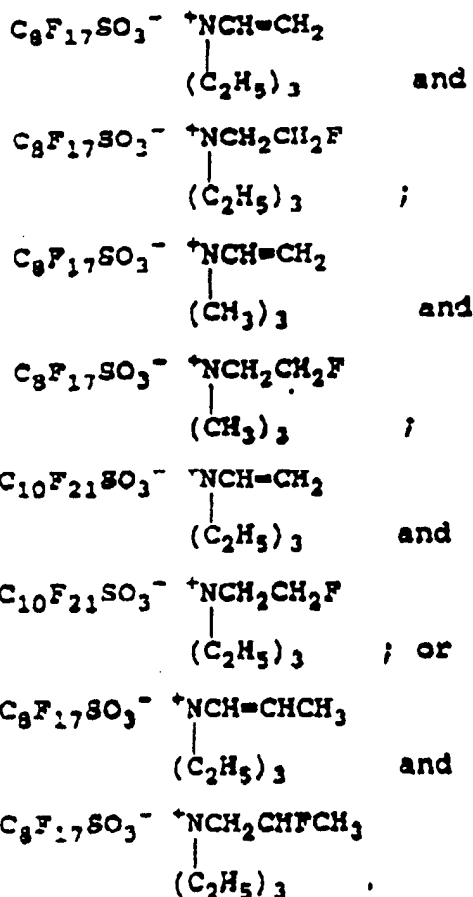


or (b) a mixture of fluorochemical amine salts which can be represented by the formulas



wherein  $R_f$  is a perfluoroaliphatic radical containing 3 to 20 carbon atoms and which can include catenary oxygen, hexavalent sulfur, and/or trivalent nitrogen hetero atoms bonded only to carbon atoms, R is H or  $CH_3$ , and each  $R^1$  is independently an alkyl radical containing 1 to 4 carbon atoms and may also contain a hydroxyl group.

2. The composition of claim 1 wherein said mixture comprises



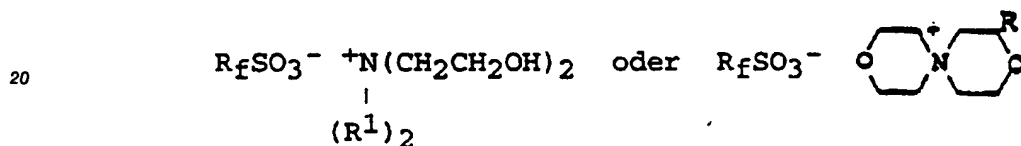
3. A process for producing fluorochemical surfactant compositions according to claim 1(b) comprising reacting (a) at least one perfluoroalkyl sulfonyl fluoride having 3 to 20 alkyl carbon atoms, (b) ethylene or propylene oxide, and (c) at least one tertiary amine,  $(R^1)_3N$ , wherein each  $R^1$  is independently an

alkyl radical having 1 to 4 carbon atoms and which may contain a hydroxyl group.

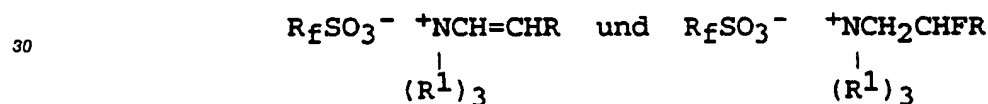
4. A process for producing fluorochemical surfactant compositions according to claim 1(a) comprising reacting (a) at least one perfluoroalkyl sulfonyl fluoride having 3 to 20 alkyl carbon atoms, (b) ethylene or propylene oxide, and (c) a trialkanol amine, the alkanol groups of which have 1 to 4 carbon atoms.
5. A process for producing fluorochemical surfactant compositions according to claim 1(a) comprising reacting (a) at least one perfluoroalkyl sulfonyl fluoride having 3 to 20 alkyl carbon atoms, (b) ethylene or propylene oxide, and (c) a dialkylalkanol amine, the alkyl and alkanol groups of which have 1 to 4 carbon atoms.

### Patentansprüche

1. Fluorchemische Tensidzusammensetzungen, umfassend (a) mindestens ein fluorchemisches Aminsatz, das durch die Formel dargestellt werden kann:

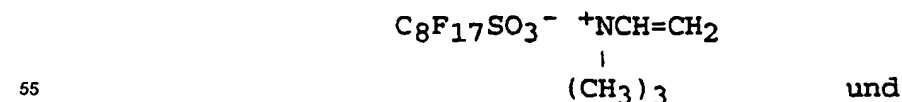
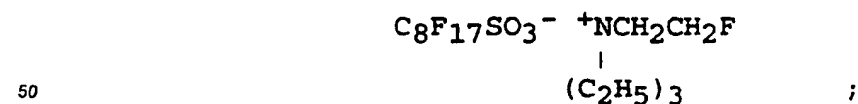
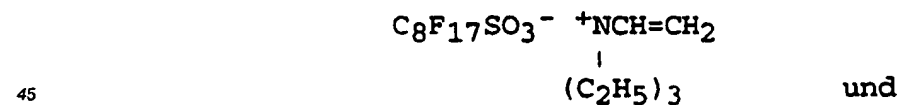


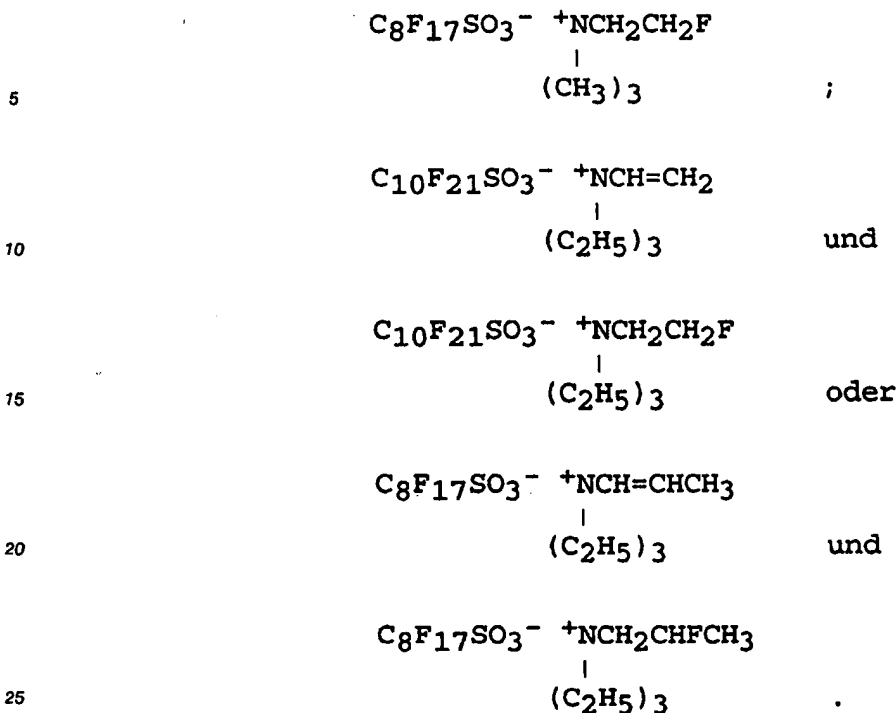
- oder (b) eine Mischung von fluorchemischen Aminsätzen, die durch die Formeln dargestellt werden können:



- darin sind  $\text{R}_f$  ein perfluoraliphatisches Radikal mit 3 bis 20 Kohlenstoffatomen, das Kettensauerstoff, sechswertigen Schwefel und/oder lediglich an Kohlenstoffatomen gebundene dreiwertige Stickstoff-Heteroatome umfassen kann; R Wasserstoff oder  $\text{CH}_3$ , wobei jedes  $\text{R}^1$  unabhängig ein Alkylradikal mit 1 bis 4 Kohlenstoffatomen ist und ebenfalls eine Hydroxylgruppe enthalten kann.

2. Zusammensetzung nach Anspruch 1, bei welcher die Mischung umfaßt:





- 30 3. Verfahren zur Herstellung fluorchemischer Tensidzusammensetzungen nach Anspruch 1 (b), umfassend das Umsetzen (a) von mindestens einem Perfluoralkylsulfonylfluorid mit 3 bis 20 Alkyl-Kohlenstoffatomen, (b) Ethylen- oder Propylenoxid und (c) mindestens einem tertiären Amin,  $(\text{R}^1)_3\text{N}$ , worin jedes  $\text{R}^1$  unabhängig ein Alkylradikal mit 1 bis 4 Kohlenstoffatomen ist und das eine Hydroxylgruppe enthalten kann.
- 35 4. Verfahren zur Herstellung fluorchemischer Tensidzusammensetzungen nach Anspruch 1 (a), umfassend das Umsetzen (a) von mindestens einem Perfluoralkylsulfonylfluorid mit 3 bis 20 Alkyl-Kohlenstoffatomen, (b) Ethylen- oder Propylenoxid und (c) einem Trialkanolamin, dessen Alkanolgruppen 1 bis 4 Kohlenstoffatome aufweisen.
- 40 5. Verfahren zur Herstellung von fluorchemischen Tensidzusammensetzungen nach Anspruch 1 (a), umfassend das Umsetzen (a) von mindestens einem Perfluoralkylsulfonylfluorid mit 3 bis 20 Alkyl-Kohlenstoffatomen, (b) Ethylen- oder Propylenoxid und (c) einem Dialkanolamin, dessen Alkyl- und Alkanol-Gruppen 1 bis 4 Kohlenstoffatome aufweisen.

#### 45 Revendications

1. Compositions de tensioactif fluorochimique comprenant :
- (a) au moins un sel d'amine fluorochimique pouvant être représenté par la formule :

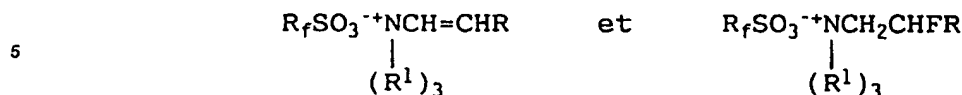
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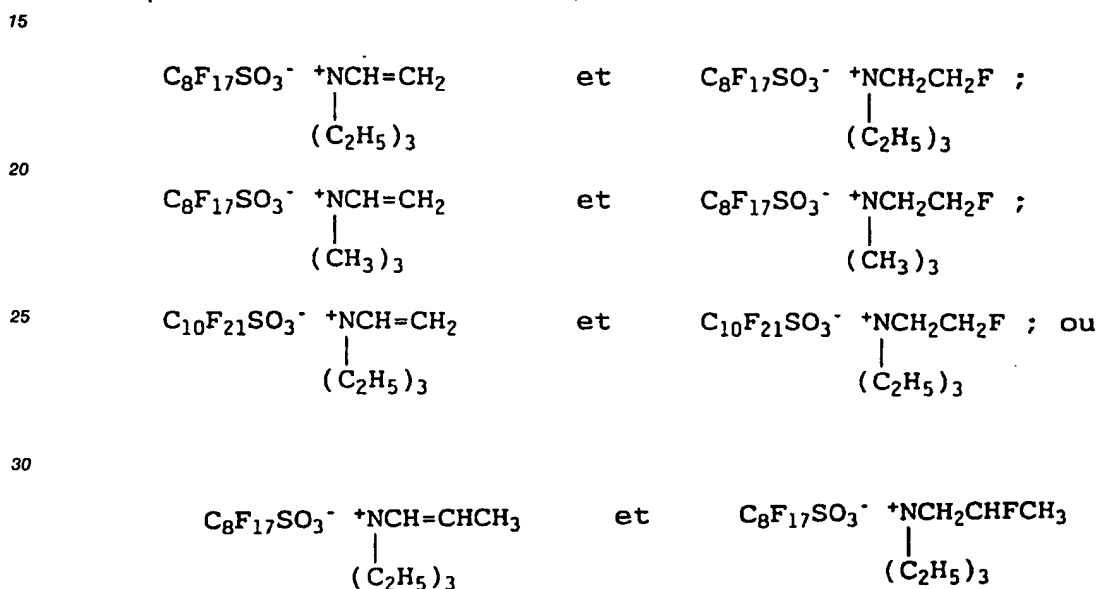
ou bien

(b) un mélange de sels d'amine fluorochimiques pouvant être représentés par les formules :



où  $\text{R}_f$  est un radical perfluoraliphatique contenant de 3 à 20 atomes de carbone et peut comprendre des hétéroatomes caténaux oxygène, soufre hexavalent et/ou azote trivalent liés uniquement à des atomes de carbone, R est H ou  $\text{CH}_3$ , et chaque radical  $\text{R}^1$  est, indépendamment des autres, un radical alkyle contenant de 1 à 4 atomes de carbone et pouvant aussi contenir un groupe hydroxyle.

2. Composition selon la revendication 1, dans lequel ledit mélange comprend :



3. Procédé pour produire des compositions de tensioactif fluorochimique selon la revendication 1 (b), qui consiste à faire réagir :

- (a) au moins un fluorure de perfluoralkylsulfonyle ayant de 3 à 20 atomes de carbone,
- (b) de l'oxyde d'éthylène ou de propylène, et
- (c) au moins une amine tertiaire  $(\text{R}^1)_3\text{N}$ , chaque radical  $\text{R}^1$ , indépendamment des autres, étant un radical alkyle comportant de 1 à 4 atomes de carbone et pouvant contenir un groupe hydroxyle.

4. Procédé pour produire des compositions de tensioactif fluorochimique selon la revendication 1 (a), qui consiste à faire réagir :

- (a) au moins un fluorure de perfluoralkylsulfonyle ayant de 3 à 20 atomes de carbone,
- (b) de l'oxyde d'éthylène ou de propylène, et
- (c) une trialcanolamine, dont les groupes alcool ont de 1 à 4 atomes de carbone.

5. Procédé pour produire des compositions de tensioactif fluorochimique selon la revendication 1 (a), qui consiste à faire réagir :

- (a) au moins un fluorure de perfluoralkylsulfonyle ayant de 3 à 20 atomes de carbone,
- (b) de l'oxyde d'éthylène ou de propylène, et
- (c) une dialkylalcanolamine dont les groupes alkyle et alcool ont de 1 à 4 atomes de carbone.